An investigation on thermal and chemical behavior of jute/hemp/flax fiber reinforced woven composites and its hybrids

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ABSTRACT

Natural fiber has emerged as a viable alternative to synthetic fibers like glass, carbon, and Kevlar for the development of polymeric composites. Present study focused on Thermo-gravimetric analysis (TGA), Differential thermal analysis (DTA), and Fourier transform infrared spectroscopy (FTIR) of the reinforced fibers and developed composites. HR-X-Ray Diffraction of neat epoxy, jute, hemp, and flax fibers was also performed. For TGA, as the temperature increases up to 250^0C, thermal degradation of all composites is higher as compared to the neat epoxy. Addition of natural fibers as reinforcement with epoxy matrix affects the transmittance peaks between 1000-1500 cm^{-1} and 1608-1738 cm^{-1} in FTIR spectra. The peaks transmittance between 1000-1500 cm^{-1} represents the chemical compositions of the fibers (hemicellulose, cellulose, lignin, and pectin) which are the necessary part of plant fibers. In X-ray diffraction, two sharp peaks appear at a diffraction angle of 21.4^0 and 14.8^0 for jute, hemp, and flax fibers. Peak at a diffraction angle (2Θ) of 26.3^0 represents α-cellulose and 14.26^0 represents non-cellulose material such as hemicellulose and lignin in fiber.

Keywords: FTIR, TGA, X-ray diffraction, hemicellulose, lignin, pectin, spectroscopy

INTRODUCTION

Natural fibers (Jute, hemp, sisal, linen, banana, flax, etc.) are eco-friendly and non-toxic, and can be used as a reinforcement to replace and reduce the use of synthetic fibers (glass, carbon, Kevlar ,aramid etc.) for manufacturing of polymeric materials.

Synthetic fibers are very harmful to the environment (Chaudhary et al., 2018b, 2018a; Das, Chaudhary, et al., 2020; Das et al., 2021). Natural fiber reinforced polymer composites are used natural fibers as reinforcement for manufacturing of polymeric composites (Bajpai et al., 2017; Chaudhary & Ahmad, 2020). In polymer composites, reinforcement strengthens the composite and matrix surrounds the reinforced fibers.

Reinforced fibers are tightly bonded to the polymer matrix, and the resulting composite is strong enough to withstand up-to their specific strength (Das, Rao, et al., 2020; Das & Chaudhary, 2020; Manral et al., 2020). Massive properties of developed composites like lightweight, high specific strength, traditional cellular structure, and solid acoustic and thermal insulation properties withstand them in various engineering applications. It is very important to predict how these composites will perform under various environmental conditions before using them. An engineer's ability to characterize these composites aids in the selection of the best composite for a given application. TGA, DTA, XRD, and FTIR are some of the experimental analysis used to study the thermal and chemical behavior of the developed composites. TGA is used to investigate the temperature-related alterations in composites' physical and chemical properties. TGA is often used to calculate the temperature at which the composites developed degrades (Guleria et al., 2017). For testing, powder samples of 5-20 mg are used. The TGA setup consists of a small electrically heated furnace and a weighing unit. As the sample in the oven is heated at a constant rate, the system continuously weighs it (Berthet et al., 2017; Dayo et al., 2017; Julkapli & Akil, 2010). FTIR spectroscopy is employed, and the Fourier transformation is used as a technique to convert raw data from the spectroscope into image format. A graph of transmittance vs. wavenumber summarizes the spectroscopic results.(Essabir et al., 2016; Lomelí-Ramírez et al., 2014).

Study on TGA, XRD study, and FTIR spectroscopy of NFRPC has been carried out by various authors. (De Rosa, Kenny, et al., 2010) studied the chemical structure of the orka fibers using FTIR. The absorbance peak of orka fibers clearly showed the presence of chemical compositions such as cellulose, hemicellulose and lignin. Authors concluded that the absorbance peak between 3600 cm^{-1} to 3100 cm^{-1} corresponds to the O-H stretching vibration and hydrogen bond of the hydroxyl group. The absorbance peaks at 1743 cm^{-1} correspond the carbonyl C=O stretching vibration of carboxylic acid in lignin and 1384 cm^{-1} to 1243 cm^{-1} belongs to the C-O stretching vibration of the acetyl group in lignin and hemicellulose component. Saha et al.(Saha et al., 2010) conducted X-RD and FTIR analysis of jute fibers. The authors concluded that jute fibers displayed cellulose crystallites justified by X-ray

diffractograms and jute fibre FTIR spectra showed a strong peak of absorbance at 3200 to 3600 cm-1, reflecting the hydrogen bond O-H stretching.

This study focused on the HR-X-Ray diffraction of neat epoxy, jute, hemp, and flax fiber. Various tests such as TGA, DTA, and FTIR for neat epoxy, as well as all the developed composites, were undertaken in this present study the thermal and chemical behavior of NFRPCs.

EXPERIMENTAL METHODS

Fibers and matrix

Jute, hemp, and flax fibers were used to develop the polymer composites. J3P grade epoxy resin and universal 10000 grade hardener are employed as the polymer matrix.

Processing Method

All the samples of composites were fabricated by closed mod hand-lay-up technique. A mild steel closed mold with dimensions of 200 x 200 mm was used. Each type of composite was made with a quarter (25%) of a percent of fibers by weight. For hybrid composites same weight percentage of fibers were used. Abbreviations of developed composites used in this study are listed in **Table 1.**

Table 1. Abbreviations of the developed composites.

Abbreviation	Description
J/Epoxy	Jute/epoxy
H/Epoxy	Hemp/epoxy
F/Epoxy	Flax/epoxy
J/H/Epoxy	Jute/hemp/ epoxy
H/F/Epoxy	Hemp/flax/ epoxy
J/H/F/Epoxy	Jute/hemp/flax/epoxy

Thermogravimetric   *analysis (TGA)*

TGA is an analytical tool that assesses the thermal stability of a material and its volatile component fraction by analyzing the shift in weight that occurs when a sample is heated at a constant rate. This method was performed by S11 Perkin Elmer Diamond TG/DTA setup in a nitrogen environment. Samples in solid pallets were examined in a nitrogen environment to a maximum temperature of 630 ^{0}C at the rate of 10 ^{0}C/min. The loss in weight was measured with an increase in temperature.

Differential thermal analysis (DTA)

DTA was carried out using DTA S11 Perkin Elmer Diamond TG/DTA setup. Samples in solid pallet form were examined in a nitrogen environment to a maximum temperature of 630 ^{0}C at the rate of 10 ^{0}C/min. The heat absorbed or released was measured along with the increase in temperature.

FTIR Spectroscopy

The FTIR (Fourier transform infrared) spectrometer from Thermo Scientific was used to perform the infrared spectroscopy (Nicolet 380). Both composites containing natural fiber were crushed into extremely fine particles. The pallets were made by combining sample particles with KBr and then pressing them into a 1 mm thick disk, as shown in **figure 1**. SEM images of fibers and their natural fiber mats for the J/Epoxy, H/Epoxy, and F/Epoxy composites developed in **Figure 2**
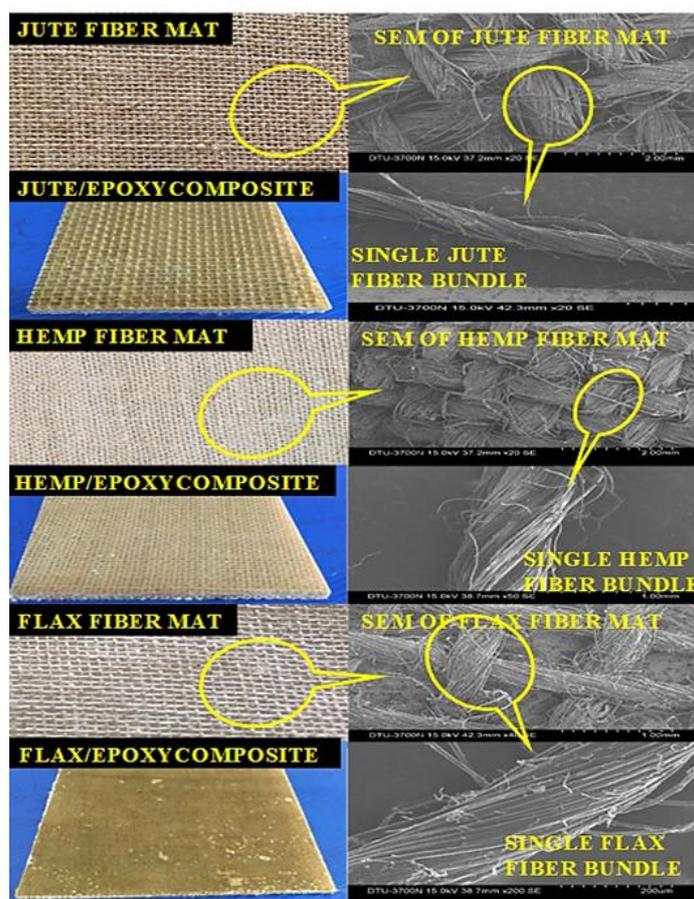

Figure 1 Pallet of epoxy mixed with KBr.

High Resolution XRD

High resolution XRD was performed by D8 Discover HR-XRD. Neat epoxy, jute, hemp, and flax fiber were crushed into small particles. Diffraction intensity of neat epoxy, jute, hemp, and flax fiber was recorded at a diffraction angle (2Ө) of 5 to 65^0 at a scan rate of 40 min.

Figure 2. J/Epoxy, H/Epoxy and F/Epoxy composites and their SEM images.

RESULT & DISCUSSIONS

Thermogravimetric analysis (TGA)

The control factor in the fabrication of polymeric materials is thermal stability. Thermogravimetric analysis was used to investigate the thermal stability of neat epoxy and all of the formed composites (TGA). The temperature range for the experiment was 35^0 C to 450^0 C. The high-temperature degradation behavior of formed polymer composites was studied using the TGA curves obtained thus far. **Figure 3** shows weight loss percentages versus degradation temperatures for neat epoxy, and all developed composites. **Table 2** shows the weight loss up to 100^0C, 200^0C, 300 to 400^0C and the final residual left.

For all the composites developed, as the temperature increases upto 250^0C, thermal degradation of composites is higher as compared to neat epoxy. Initial degradation occurs between the temperature range of 35^0C to 250^0C because of vaporization of moisture content present in the fibers (De Rosa, Santulli, et al., 2010). From temperature 35^0C to 250^0C, all the developed composites including neat epoxy showed an almost similar pattern of degradation.

But after 250^0C, suddenly more degradation occured in all composites and neat epoxy. In composites, the initial rate of degradation is higher because of the decomposition of hemicellulose, cellulose, lignin and pectin present in jute, hemp and flax fibers(Guleria et al., 2017; Julkapli & Akil, 2010; Revati et al., 2017) . For neat epoxy initially 0.82 wt. % of the original weight is reduced at 100^0C. As natural fibers are hydrophilic in nature, they absorb a large amount of water as compared to neat epoxy. At initial stage (35 ^{0}C to 250^0C), weight loss of neat epoxy is less than 8% while all the developed composites lost more than 8% of its original weight. But in the second stage, from temperature 250^0C to 400^0C all the developed composites degrades swiftly as well as neat epoxy and loses 70% of its original weight. In the 2nd stage, the thermal degradation of neat epoxy is higher (67.58%) as compared to all developed composites. In 2nd stage degradation occurs due to the volatile content in the polymer matrix and debasement of epoxy polymer (Kumar et al., 2014). At temperature, above 250^0C, the composite structure showed lower degradation than neat epoxy. The thermal stability of the developed composites is higher as compared to the neat epoxy. This increased stability of composites compared to neat epoxy is due to improved fiber–matrix interaction. A similar study was conducted by Sreenivasan et al.(Sreenivasan et al., 2015). Authors studied weight loss of Sansevieria-cylindrica/polyester composite under varying temperature with the help of thermogravimetric analysis. Authors concluded that major weight loss of the developed composite was noticed between 250^0C to 400^0C due to breakage of the internal structure of composite by thermal loading. The composite degradation at higher temperatures is protected by strong interfacial adhesion between the fiber-matrix interface.

At temperature 450^0C, the residual weight of neat epoxy is 7 wt. % of the original weight, present in the form of charcoal, carbon and ash content left. For the jute/epoxy composite, initial weight reduction is 2.32 wt. % of original weight from temperature 35 to 100^0C, that shows an increment in weight degradation as compared to neat epoxy. In the next phase 6.53 wt. % of the original weight is reduced at 200 ^{0}C. The dominant degradation of 63.84 wt. % of original weight occurs at 310 to 400^0C. At a temperature of 499 ^{0}C, final residual weight is 8.28 wt. % of the original weight. For hemp/epoxy composite initially 3.48 wt. % of original weight degrades at 100^0C. At 200 ^{0}C, 5.8 wt. % of the original weight is reduced for hemp/epoxy composite.

The extensive degradation of hemp/epoxy composite occurs at a temperature range of 290^0C to 380^0C which is 65.68 wt. % of the original weight. The final degradation occurs at a temperature of 422^0C and the residue is 12.52 wt. % of the original weight. For flax/epoxy composite, 2.38 wt. % of the original weight is reduced at 100 ^{0}C that is almost similar to jute/epoxy composite. Degradation of 5.42 wt. % of the original weight occurs at a temperature of 200 ^{0}C and 57.3 wt. % of original weight

occurs at a temperature of 300 ^{0}C to 400 ^{0}C. At 438 ^{0}C, the final residue is 25 wt. % of the original weight. The final residue contains carbon content, ash content and other carbon-based material.

For jute/hemp/epoxy composite, 2.71 wt. % of the original weight is reduced at 100 ^{0}C. Second degradation of 2.31 wt. % of original weight occurs at a temperature of 200 ^{0}C. The major weight reduction occurs at a temperature of 320 to 410 ^{0}C, that is 46.72 wt. % of the original weight. At 438 ^{0}C, the final residue is 38 wt. % of the original weight. For hemp/flax/epoxy composite, thermal degradation curve shows that the initial weight loss up to 100 ^{0}C is 3.73 wt. % of the original weight and this is almost similar to the hemp/epoxy composite. The second degradation starts after 200 ^{0}C and the loss of mass is 6.64 wt. % of the original weight. The extensive degradation of 61.6 wt. % of original weight occurs between 300^{0}C to 400^{0}C. Final mass residue at 424^{0}C is 6.86 wt. % of the original weight. Thermal degradation curve of jute/hemp/flax/epoxy composite shows that 4.98 wt. % of the original weight was lost up to a temperature of 100 ^{0}C. Further degradation of 7.95 wt. % of original weight occurs up to a temperature of 200^{0}C. The Final mass residue is 17.33 wt. % of original weight containing charcoal and ash content. TGA of J/H/F/Epoxy composites shows that the weight loss between the temperature range (200 to 400^{0}C) occurs due to the decomposition of hemicellulose, cellulose, lignin and pectin present in jute, hemp and flax fibers.

Table 2 Thermal degradation(TD) of composites at different temperature range.

| Composites	Weight Loss(%)			
	Upto 100^{0}C	100 to 250^{0}C	250 ^{0}C to 400^{0}C	Final residual
Neat epoxy	0.820	6.318	67.580	7.000
Jute/Epoxy	2.324	6.534	63.841	8.280
Hemp/Epoxy	3.486	5.809	65.680	12.510
Flax/Epoxy	2.385	5.424	57.300	25.000
Jute/Hemp/Epoxy	2.710	2.312	46.720	38.000
Hemp/Flax/Epoxy	3.740	6.640	61.600	6.860
Jute/Hemp/Flax/Epoxy	4.980	7.950	64.200	7.330
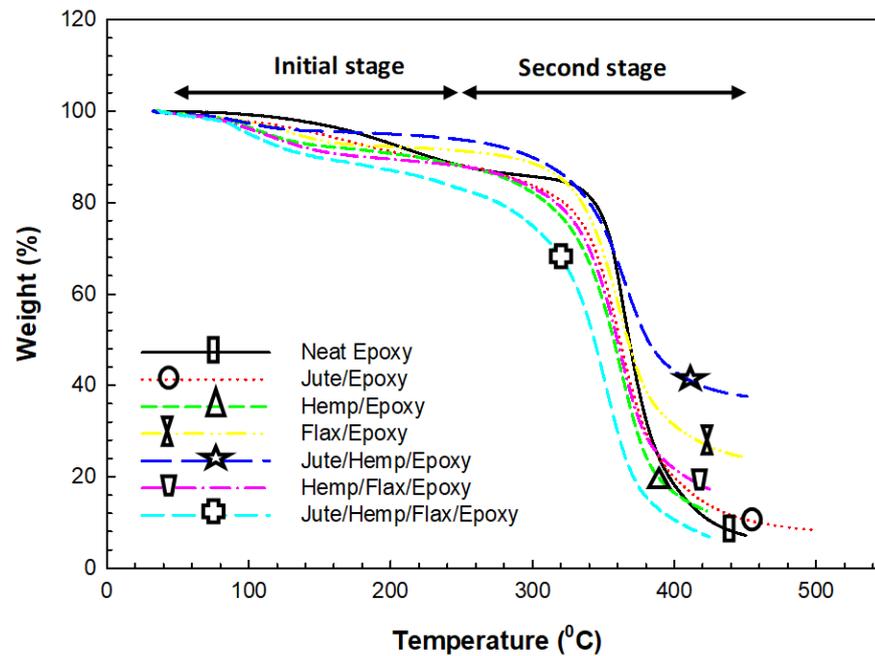
Figure 3 Thermogravimetric analysis of neat epoxy and composites.

Differential thermal analyzer (DTA)

The differential thermal analyzer is used to determine the nature of reaction during polymerization (either endothermic or exothermic). The temperature range used for the analysis is 35–450 ^{0}C. **Figure 4** shows DTA results of developed composites with neat epoxy resin.

DTA curve indicates the direction of reactions which may be either exothermic or endothermic. **Table 3** shows the amount of heat absorbed and the softening temperature of the developed composites. Results showed that all the developed composites absorbed heat with an increase in temperature. The maximum amount of heat absorption at a glass transition temperature of jute/epoxy composite showed a better heat-absorbing capacity than other developed composites. H/Epoxy and H/F/Epoxy composites absorbed almost equal heat of 30 mW\mg and 30.83 mW\mg at glass transition temperature of 357^{0}C and 356.2^{0}C. J/H/Epoxy and J/H/F/Epoxy composites absorbed the heat of 17.02 mW\mg and 28.33 mW\mg at a glass transition temperature (T_g) of 372.9 ^{0}C and 354 ^{0}C respectively.
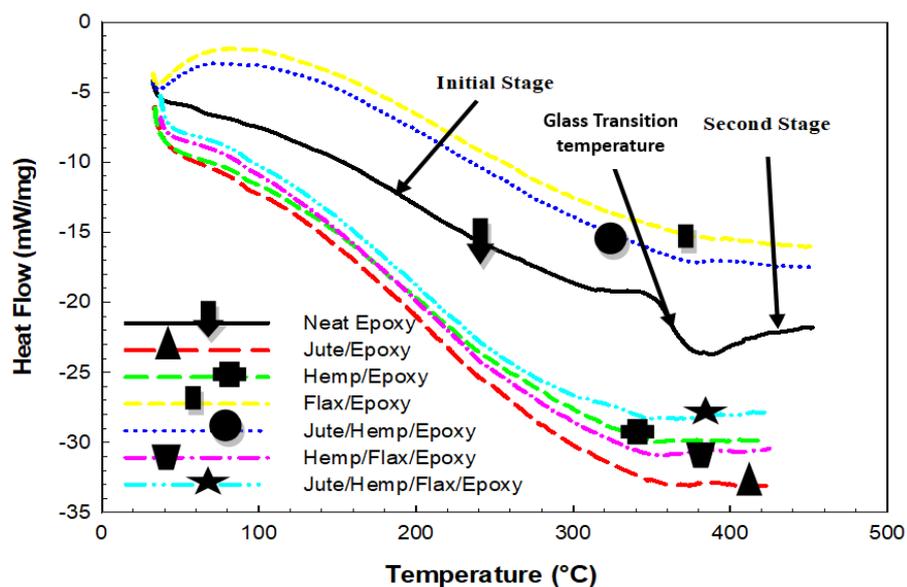
**Figure 4** Differential thermal analysis of neat epoxy and composites.

Table 3 Heat absorbed by the composites at Glass Transition Temperature (T_g).

Composites	Maximum heat absorbed (mW\mg)	Glass transition temperature (^{0}C)
Neat epoxy	17.33	316
Jute/epoxy	33.02	357.3
Hemp/epoxy	30.0	357.0
Flax/epoxy	15.42	378.0
Jute/hemp/epoxy	17.08	372.9
Hemp/flax/epoxy	30.83	356.2
Jute/hemp/flax/epoxy	28.33	354.0

Fourier transform infrared spectroscopy (FTIR)

Infrared transmittance peaks recorded for the developed composites are compared with neat epoxy as shown in **figure 5**. Jute, hemp and flax fibers are plant-based fibers having a chemical composition consisting of cellulose, hemicellulose, lignin and pectin(Mwaikambo & Ansell, 2002). With Addition of natural fibers (jute, hemp, and flax) with epoxy resin changes the bonding structure at the mating surface of fibers and matrix which influences the infrared transmittance peaks at different wavenumbers. Absorbance peaks between the wavenumber of 500-4000 cm^{-1} are analyzed. All the

developed composite shows a similar pattern of wavenumbers with different values of absorbance peaks. The absorbance peaks between the wavenumber of 1000-1500 cm^{-1} which indicate the presence of hemicellulose, cellulose, lignin, and pectin which are the important constituent part of natural (plant) fibers(Mwaikambo & Ansell, 2002). The FTIR spectra of J/Epoxy composite shows the clear fluctuation in the intensity of peak between the wavenumber of 1608 cm^{-1} to 1738 cm^{-1}, which indicates the presence of fatty acids in jute fibers as compared to hemp and flax fibers. Peaks between the wavenumber of 2927 cm^{-1} to 3427 cm^{-1} seen in all developed composites show the formation of hydrogen bonding and presence of hydroxyl group in all the developed composites (Sgriccia et al., 2008). Absorbance peaks of neat epoxy between the wavenumber of 1000-1500 cm^{-1} are absent, which shows the absence of hemicellulose, cellulose, lignin, and pectin. The absorbance peak of neat epoxy between the wavenumber of 3200 cm^{-1} to 3600 cm^{-1} indicates the hydrogen bonding and presence of hydroxyl groups(Sawpan et al., 2011). The various author carried out their study on the FTIR spectroscopy of natural fiber reinforced polymer (FRP) composites. Mwaikambo and Ansell(Mwaikambo & Ansell, 2002) experimentally studied the FTIR spectra of jute, hemp, sisal & kapok fibers. Authors concluded that the absorbance peak between 1000cm^{-1} to 1500 cm^{-1} indicates the presence of hemicellulose and lignin in plant-based fibers and absorbance peak between the wavenumber of 3200 cm^{-1} to 3600 cm^{-1} indicates the hydrogen bonding and the presence of hydroxyl groups. Sgriccia et al.(Sgriccia et al., 2008) investigated the FTIR spectrum of hemp and kanaf fibers. Authors concluded that the lignin peak at a wavenumber of 1500 cm^{-1} is present in kenaf fibers and CH stretch is also present at a wavenumber of 2900 cm^{-1} in kenaf and hemp fibers. Sawpan et al.(Sawpan et al., 2011) investigated the FTIR spectra of hemp fiber. Authors concluded that transmittance peak at a wavenumber of 3410 cm^{-1} shows hydrogen bonding and presence of hydroxyl groups. Satish kumara et al.(Sathishkumar et al., 2013) investigated the FTIR spectrum of sansevieria fiber. Authors concluded that the transmittance peak between the 400 to 1500 cm^{-1} wavenumber indicates the compositions dependent on plant fiber (cellulose, hemicellulose, and lignin) and the sharp peak between the 3100 cm^{-1} to 3600 cm^{-1} wavenumber reflects the bonding of hydrogen as well as the presence of hydroxyl groups.
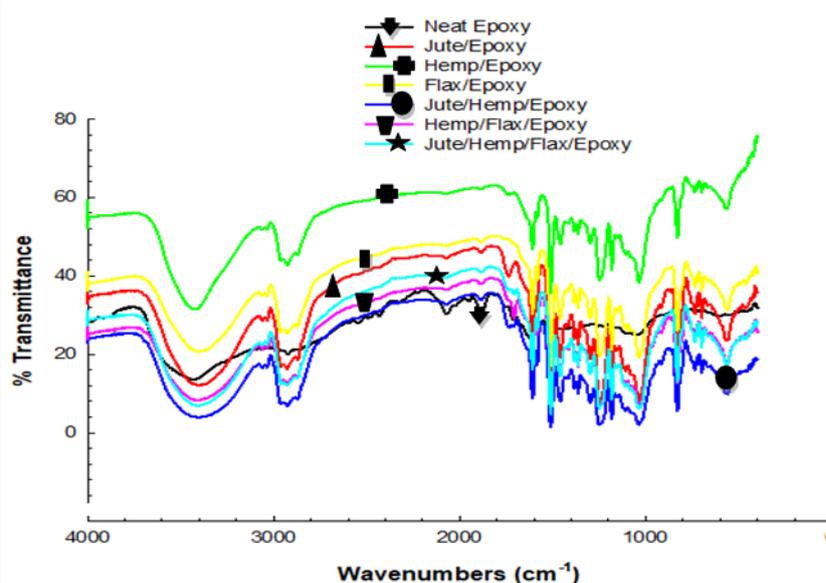
Figure 5 FTIR spectra of neat epoxy and composites.

High Resolution X-Ray Diffraction (HR-XRD)

Figure 6 shows the X-Ray Diffractograms of natural fibers and neat epoxy. The pattern of diffraction angle with different intensities is similar for jute, hemp, and flax fibers. The same pattern X-ray diffraction was recorded by bajpai et al.(Bajpai et al., 2013) for grevia optiva, nettle and sisal. It is observed from the diffractograms of jute, hemp, and flax fibers that there are two major crystalline peaks occur for each natural fiber. The primary largest crystalline peak occurs at a diffraction angle of 21.4^0 and a second sharp peak of 14.8^0 for jute, hemp, and flax fibers. For neat epoxy, a single and clear peak was observed at a diffraction angle (2Θ) of 17.5^0 as a clear indication of more crystalline material. If the material is amorphous in nature with a high content of its constituent materials such as cellulose, hemicellulose, lignin, and pectin then the peaks in the diffractograms occurs with low intensity and therefore the sharpness of the peak reduces and become filthy(Jayaramudu et al., 2010). Various author carried out their study on the X-ray diffraction analysis of natural fiber reinforced polymer composites. Sawpan et al.(Sawpan et al., 2011) performed XRD analysis of hemp fiber. Authors concluded that the hemp fiber showed the sharp peak of cellulose at a diffraction angle (2Θ) of 22^0. Satish kumar et al. (Sathishkumar et al., 2013) investigated the X-ray diffractograms of sansevieria fiber. Authors concluded that two diffraction peaks found at a diffraction angle (2Θ) of 26.3^0 and 14.26^0. The peak at a diffraction angle (2Θ) of 26.3^0 represents of α-cellulose and 14.26^0 represents noncellulose material such as hemicellulose and lignin in a natural fiber.
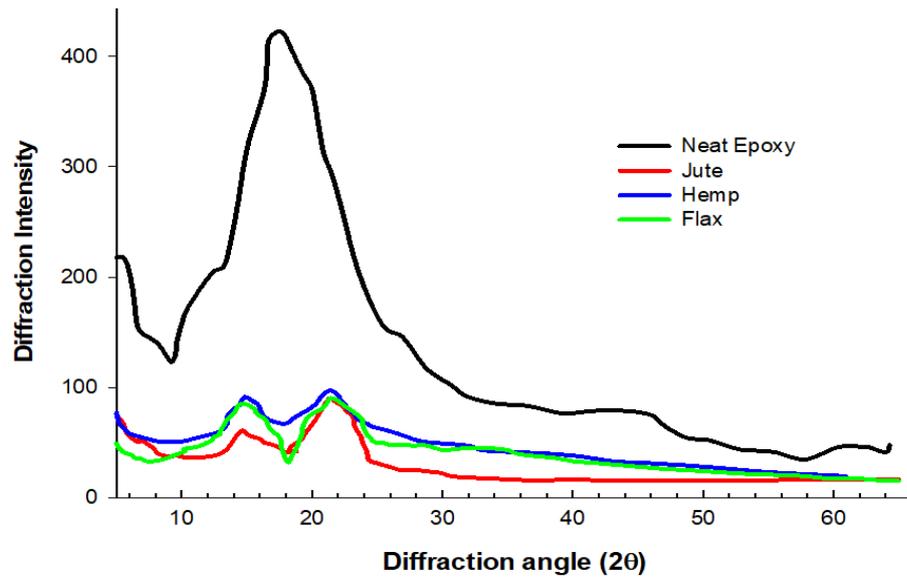
Figure 6 X-ray diffraction spectra of neat epoxy, jute, hemp, and flax fibers.

Percentage of crystallinity and crystallinity index was calculated using the given equations:

$$\text{Percentage of Crystallinity} = \frac{I_{21}}{I_{21} + I_{14}} \times 100 \tag{1}$$

$$\text{Crystallinity Index} = \frac{I_{21} - I_{14}}{I_{21}} \times 100 \tag{2}$$

Where I_{21} and I_{14} are the corresponding crystalline and amorphous diffraction peaks near to the 21 and 14 angles (2 Theta). For neat epoxy, jute, hemp and flax, the percentage of crystallinity and crystallinity index are shown in **table 4**.

XRD analysis shows that the fabricated composites are neither pure crystalline nor pure amorphous in nature and therefore, these are semi-crystalline in nature. Crystallinity index indicates the amount of cellulose present in the developed polymer composites (Sawpan et al., 2011). For a higher value of crystallinity index, more would be the cellulose content in the fiber (Reddy et al., 2013; Seki et al., 2013). The crystallinity index of flax fiber is 6.4, which is lower than jute and hemp fiber. A lower value of the crystallinity index of flax fiber indicates the lower percentage of cellulose as compared to jute and hemp fiber. The crystallinity index of jute fiber is 59%, which is higher in comparison to hemp and flax fiber. According to the higher crystallinity index, jute fiber is more crystalline in comparison to hemp and flax fiber.

Table 4 % crystallinity and crystallinity index of the jute, hemp, & flax fiber.

Sample	% of Crystallinity	% of crystallinity Index
Jute	59.2	31.2
Hemp	52.2	6.4
Flax	51.6	8.57

CONCLUSIONS

TGA and FTIR spectroscopy of all developed composites, as well as X-ray diffraction of neat epoxy and natural fibers(jute, hemp, and flax), were investigated in this work . The following are some of the notable results from this investigation:

1. Temperature range used for thermogravimetric analysis was 35^0C to 450^0C. For all the developed composites, with an increase in temperature upto 250^0 C, thermal degradation of composites is higher as compared to neat epoxy.
2. In TGA, initial degradation occurs between the temperature ranges of 35^0C to 250^0C, which is because of the vaporization of moisture present in the fibers.
3. Addition of natural fibers with resin (epoxy) also the influenced infrared transmittance peaks at different wavenumbers in FTIR spectrum in comparison to FTIR spectra of neat epoxy.
4. In FTIR spectra, the absorbance peaks between the wavenumber of 1000-1500 cm^{-1} clearly indicate the presence of hemicellulose, cellulose, lignin, and pectin which are a necessary part of plant-based fibers. Peaks between the wavenumber of 2927 cm^{-1} to 3427 cm^{-1} represent the formation of hydrogen bonding and presence of hydroxyl group in all the developed composites and neat epoxy.
5. In X-ray diffractor-grams, two major peaks occurred at a diffraction angle of 21.4^0 and a second sharp peak of 14.8^0 for jute, hemp, and flax fibers. But, in case of neat epoxy, a single and clear peak was observed at a diffraction angle (2Θ) of 17.5^0.
6. Two major peaks of jute, hemp and flax fiber at a diffraction angle of $21.4^0$and 14.8^0represent the presence of chemical compositions for plant-based fibers like hemicellulose and α-cellulose but the single peak of neat epoxy justified the absence of hemicellulose, lignin and pectin.

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